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## CO<sub>2</sub> saturated brine injected into fractured shale: An X-ray micro-tomography in-situ analysis at reservoir conditions

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### Abstract

Fracture morphology and permeability are key factors in enhanced gas recovery (EOR) and Carbon Geo-storage (CCS) in shale gas reservoirs as they determine production and injection rates. However, the exact effect of CO<sub>2</sub>-saturated (live) brine on shale fracture morphology, and how the permeability changes during live brine injection and exposure is only poorly understood. We thus imaged fractured shale samples before and after live brine injection in-situ at high resolution in 3D via X-ray micro-computed tomography. Clearly, the fractures' aperture and connectivity increased after live brine injection.

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**Keywords:** shale; live brine; fracture morphology; permeability

### 1. Introduction

Carbon capture and storage (CCS) in underground geological formations, such as depleted oil/gas reservoirs or saline aquifers is considered to be an effective approach to trap large amounts of CO<sub>2</sub> and thus mitigate climate warming [1-3]. CO<sub>2</sub> injected into sandstone [4-6], limestone [7-9] and coal seams [10-12] has been extensively

investigated. However, shale gas reservoirs - which contributed significantly towards energy security in the past years [13-17] - have not been systemically evaluated in this context.

The injected CO<sub>2</sub> is partially miscible with the resident brine [18], and reacts with the formation water to carbonic acid, thus becoming acidic [19, 20]. This CO<sub>2</sub>-saturated (live) brine chemically reacts with the host rock [21-24]. This is particularly the case for limestone, which is significantly dissolves during live brine injection, which thus drastically increases limestone permeability [25-32]. This effect also occurs in the sandstone as some cements dissolve (such as calcite, ferrodolomite) [33-37]. It is thus clear that the acidic environment severely impacts on the micro structure and permeability of the rock.

However, no significant attention has been given to the potential structural, morphological changes in shale. We thus e imaged the shale in 3D at high resolution in-situ with X-ray micro-computed tomography before and after live brine injection to assess the micro structural characteristics and their potential changes.

## 2. Materials and experimental methodology

A plug (5 mm diameter and 5 mm length) was drilled out of a larger core sample from a shale gas reservoir in the Ordos basin in China. The plug was housed in an X-ray transparent high pressure unconstrained flow cell [28, 38], which was connected to an experimental core flooding apparatus built for fluid permeability measurement. The whole system was vacuumed for one day to remove all air from the system. All fluids were heated to 323 K and the core was subjected to 5 MPa effective stress. The core was then imaged by x-ray computerized micro-tomography (microCT) at a resolution of (3.43  $\mu\text{m}$ )<sup>3</sup>. Subsequently live brine at a constant flow rate of 0.1 mL/min was injected for almost 5 hours, and the plug was microCT imaged again.

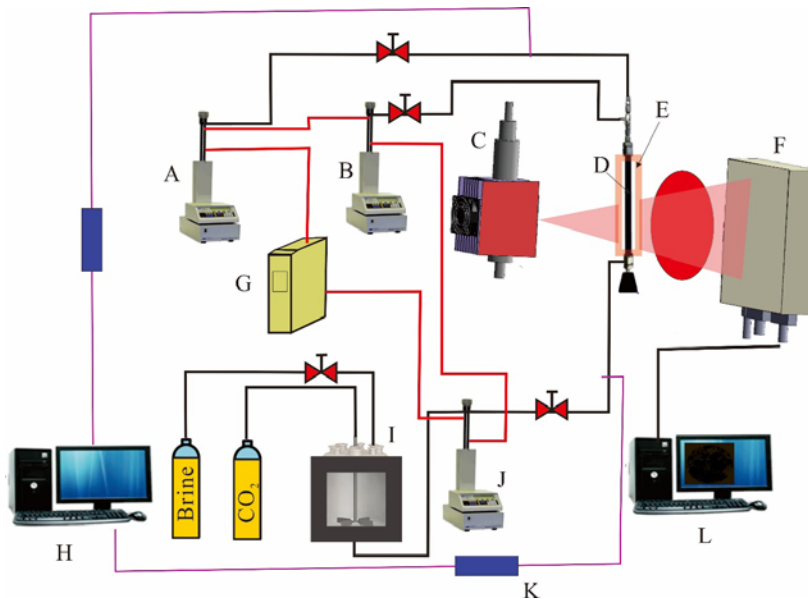


Fig. 1. (A) Injection pump, (B) Confining pump, (C) X-ray source; (D) pressurized core holder, (E) Heated tape, (F) Detector panel, (G) Water bath, (H) computer for data logging, (I) Reactor, (J) Production pump, (K) Pressure sensor, (L) CT images record computer.

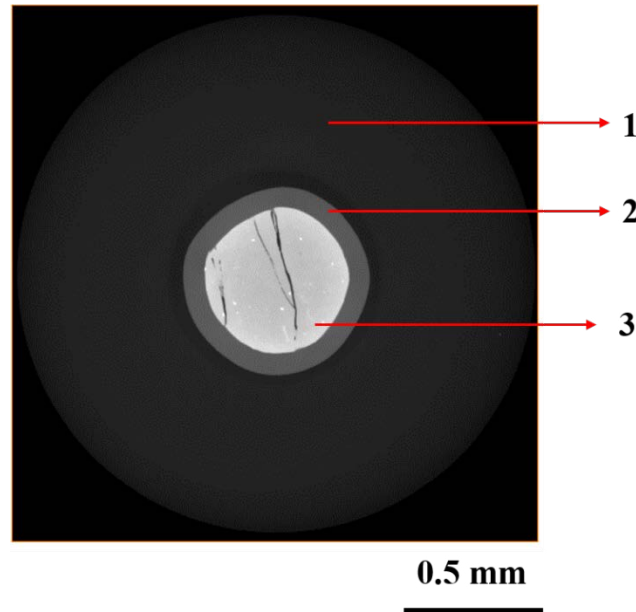


Fig. 2. Raw computerized tomography (CT) image slice showing the core holder cell (1), Teflon sleeve (2) and sample (3).

### 3. Results and discussion

#### 3.1. Morphology of fracture network

The fracture network in the shale sample is visualized in Figures 3 and 4, before and after live brine injection. The black lines are the fractures, white points are high density minerals (such as pyrite and siderite), and grey is the clay mineral matrix (Figure 4). A significant change in the fractures morphology was observed before and after live flooding. Clearly, the fracture aperture increased after live brine injection, compare also Figure 5. Importantly, the fractures' connectivity also increased after live brine injection.

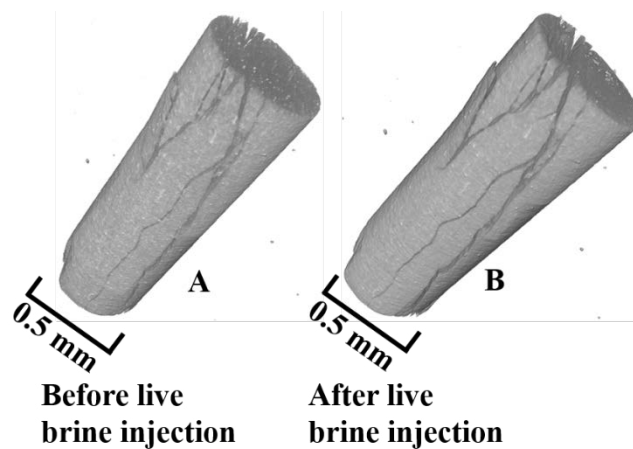


Fig. 3. 3D visualizations of the shale sample before (A) and after (B) live brine injection. The dark grey lines are the fractures, and grey is the mineral matrix.

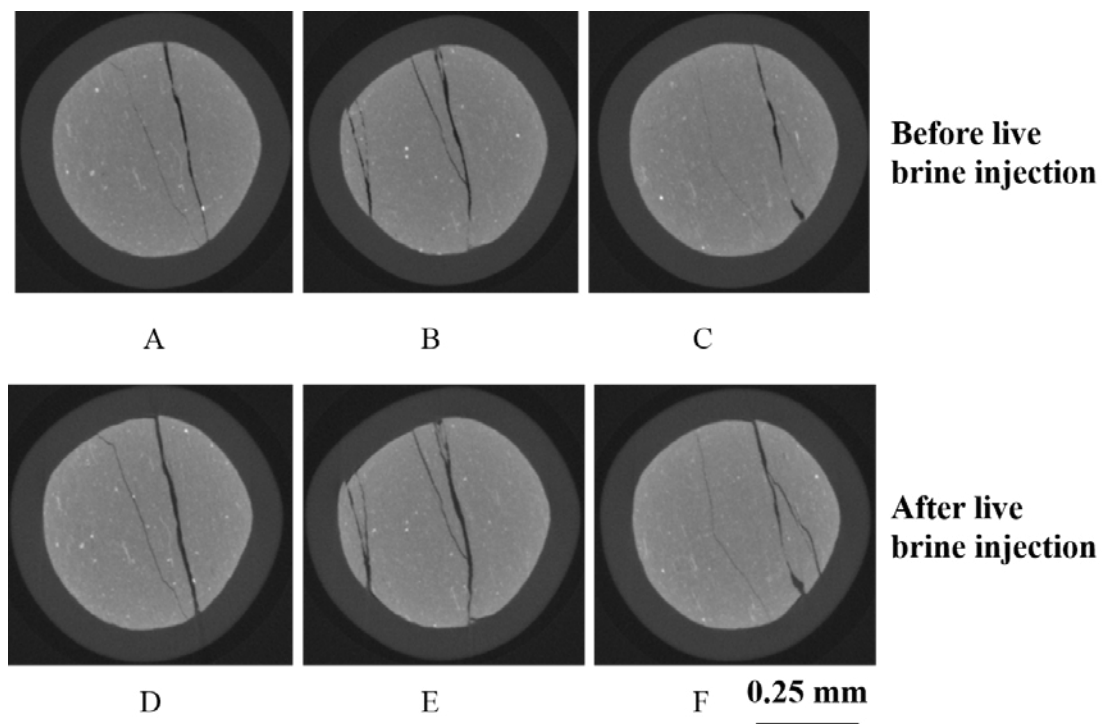


Fig. 4. 2D slices through the micro CT images of the shale sample before (a, b, c) and after (d, e, f) live brine injection.

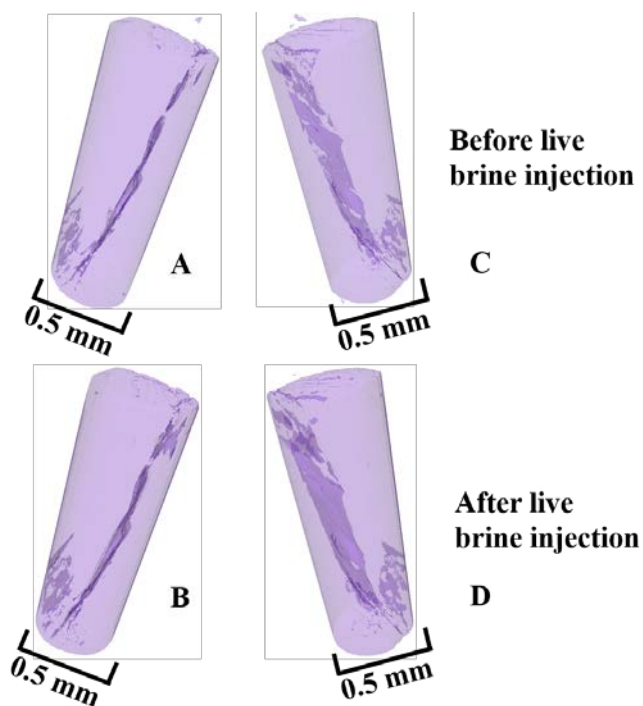


Fig. 5. Transparent 3D visualizations of the shale sample before (a, b) and after (c, d) live brine injection.

### 3.2. Discussion

This increase in fracture network size and connectivity after live brine injection can be interpreted as follows.

- (1) the hydraulic injection pressure of the fluid opened the fractures;
- (2) the acidic live brine dissolved some shale minerals, e.g. the carbonate cement [39, 40], this increased the connectivity.

### 4. Conclusion

Rock microstructure is an essential factor which determines CO<sub>2</sub> storage capacity and injectivity in shale gas reservoirs. CO<sub>2</sub> injected into shale gas reservoirs will cause adsorption, dissolution and molecular diffusion [15, 16, 41], which will in turn affect the microstructure.

In order to assess the possibility of the shale reservoirs geosequestration, we thus imaged a shale sample in high resolution via 3D microCT before and after 5 hrs live brine injection to investigate the change of the stress regime acting on the fractures. We observed that the fractures' apertures and fracture connectivity increased significantly after live brine injection. Hence, we conclude that CO<sub>2</sub> injection into shale reservoirs can cause significant morphological changes, which will affect storage efficiency.

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